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(54) Title: LIQUID COMPOSITION IN A POUCH

(57) Abstract: The present invention relates to a liquid composition comprising a transparent or translucent liquid medium and solid particles contained within the liquid medium wherein the liquid medium comprises less than 10% by weight of water and the composition is contained within a pouch made from a transparent or translucent water-soluble material, so that the individual solid particles are visible from outside the pouch. Preferably the solid particles have a mean geometric diameter of between 0.5mm and 12mm.



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LIQUID COMPOSITION IN A POUCH

Field of Invention

This invention relates to liquid composition comprising a transparent or translucent liquid medium and solid particles contained within the liquid medium.

Background to the Invention

GB-A-1 303 810, published on 24th January 1973, discloses clear, liquid compositions which comprise a visually distinct component of particle size at least 0.5mm diameter. The liquid medium in which the visually distinct components are suspended preferably has the rheological properties of a Bingham body. That is to say that by virtue of its internal structure the medium will exhibit a yield value from which it is possible to calculate the maximum size of particle which can stably be suspended for a given difference in density between the medium and suspended particles.

However if the desired size of suspended particles exceeds the size which is the maximum theoretically which can be suspended without either sinking or floating then either the liquid medium would have to be modified in order to increase the yield value or the density difference between the medium and the suspended particles would have to be decreased. Neither of these solutions may be practical and economical. Furthermore, if the yield point of the medium is too high, then it becomes difficult to pour the product.

It is an object of the present invention to provide visually appealing liquid compositions comprising suspended particles. The suspended particles themselves may either contribute to the aesthetic appearance of the product, or they may have some technical functionality, or both of these. The problems of instability, i.e. particles separating either by floating or sinking, and of excessively high yield points are overcome.

Summary of Invention

The present invention provides a liquid composition comprising a transparent or translucent liquid medium and solid particles contained within the liquid medium, wherein the liquid medium comprises less than 10% by weight of water and the composition is contained within a pouch made from a transparent or translucent water-soluble material, so that the individual solid particles are visible from outside the pouch.

Preferably the water-soluble pouch material comprises a film of water-soluble material selected from the group consisting of polyacrylates and water-soluble acrylate copolymers, methylcellulose, carboxymethylcellulose sodium, dextrin, ethylcellulose, hydroxyethyl cellulose, hydroxypropyl methylcellulose, maltodextrin, polymethacrylates and mixtures and laminates thereof. More preferably the water-soluble pouch material comprises polyvinyl alcohol (PVA).

In order to be visible to the human eye when viewed from the outside of the pouch, it is preferred that the solid particles have a mean geometric diameter of at least 0.5 millimetres, more preferably the mean geometric diameter is between about 0.5 millimetres and about 12 millimetres, and most preferably it is between about 1 millimetre and about 5 millimetres. The geometric diameter of an individual solid particle as defined herein is the diameter of a hypothetical spherical particle having the same volume as the individual particle. When more than one solid particle is considered, the mean geometric diameter is the arithmetical mean value of the geometric diameters of the individual particles.

Various industries, including agrochemicals and laundry industries have been trying to develop ways to make dosing of the products easier. One of method for this is to provide a "unit dose" enclosed within a water-soluble pouch. The composition of the present invention is preferably a unit dose of a laundry composition comprising at least anionic surfactant and fatty acid builder. One of the advantages of the present invention is that the solid particles do not necessarily need to be stably suspended in the liquid medium, but rather the solid particles may sink or float in the liquid medium. This allows much more flexibility to the formulator because it is no longer necessary to match the densities of the solid particle and the liquid medium.

Detailed Description of Invention

Pouch and Material Therefor

The pouch of the invention, herein referred to as "pouch", is typically a closed structure, made of a water-soluble film described herein, enclosing a volume space which comprises a composition. Said composition is described in more detail hereinafter. The pouch can be of any form, shape and material which is suitable to hold the composition, e.g. without allowing the release of the composition from the pouch prior to contact of the pouch to water. The exact execution will depend on for example, the type and amount of the composition in the pouch, the number of compartments in the pouch, the characteristics required from the pouch to hold, protect and deliver or release the compositions.

The pouch may have one compartment, holding the liquid composition, or it may have a number of compartment, attached to one another or non-attached to one another, thus having one compartment enclosing (but not attaching) another compartment.

The pouch may be of such a size that it conveniently contains either a unit dose amount of the composition herein, suitable for the required operation, for example one wash, or only a partial dose, to allow the consumer greater flexibility to vary the amount used, for example depending on the size and/or degree of soiling of the wash load.

It may be preferred that the water soluble film and preferably the pouch as a whole is stretched during formation and/or closing of the pouch, such that the resulting pouch is at least partially stretched. This is to reduce the amount of film required to enclose the volume space of the pouch. When the film is stretched the film thickness decreases. The degree of stretching indicates the amount of stretching of the film by the reduction in the thickness of the film. For example, if by stretching the film, the thickness of the film is exactly halved then the stretch degree of the stretched film is 100%. Also, if the film is stretched so that the film thickness of the stretched film is exactly a quarter of the thickness of the

unstretched film then the stretch degree is exactly 200%. Typically and preferably, the thickness and hence the degree of stretching is non-uniform over the pouch, due to the formation and closing process.

Another advantage of stretching the pouch, is that the stretching action, when forming the shape of the pouch and/or when closing the pouch, stretches the pouch non-uniformly, which results in a pouch which has a non-uniform thickness. This allows control of the dissolution of water-soluble pouches herein, and for example sequential release of the components of the detergent composition enclosed by the pouch to the water.

Preferably, the pouch is stretched such that the thickness variation in the pouch formed of the stretched water-soluble film is from 10 to 1000%, preferably 20% to 600%, or even 40% to 500% or even 60% to 400%. This can be measured by any method, for example by use of an appropriate micrometer. Preferably the pouch is made from a water-soluble film that is stretched, said film has a stretch degree of from 40% to 500%, preferably from 40% to 200%.

The pouch is made from a water-soluble film. The material in the form of a film can for example be obtained by casting, blow-moulding, extrusion or blow extrusion of the polymer material, as known in the art. The film may be a laminate of two or more films.

The material is water-soluble and has a solubility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out hereinafter using a glass-filter with a maximum pore size of 50 micrometers, namely:

Gravimetric method for determining water-solubility or water-dispersability of the material of the compartment and/or pouch:

50 grams \pm 0.1 gram of material is added in a 400 ml beaker, whereof the weight has been determined, and 245ml \pm 1ml of distilled water is added. This is stirred vigorously on magnetic stirrer set at 600 rpm, for 30 minutes. Then, the mixture is filtered through a folded qualitative sintered-glass filter with the pore sizes as defined above (max. 50 micrometer). The water is dried off from the collected filtrate by any conventional method, and the weight of the remaining polymer is

determined (which is the dissolved or dispersed fraction). Then, the % solubility or dispersability can be calculated.

Preferred polymer copolymers or derivatives thereof are selected from polyvinyl alcohols, polyalkylene oxides, acrylic acid, cellulose, cellulose ethers, cellulose esters, polyvinyl acetates, polycarboxylic acids and salts, polyaminoacids or peptides, polyamides, copolymers of maleic/acrylic acids, polysaccharides including starch and gelatine, natural gums such as xanthum and carragum. More preferably the polymer is selected from polyacrylates and water-soluble acrylate copolymers, methylcellulose, carboxymethylcellulose sodium, dextrin, ethylcellulose, hydroxyethyl cellulose, hydroxypropyl methylcellulose, maltodextrin, polymethacrylates, most preferably polyvinyl alcohols, polyvinyl alcohol copolymers and hydroxypropyl methyl cellulose (HPMC). Preferably, the level of a type polymer (e.g., commercial mixture) in the film material, for example PVA polymer, is at least 60% by weight of the film.

The polymer can have any weight average molecular weight, preferably from about 1000 to 1,000,000, or even from 10,000 to 300,000 or even from 15,000 to 200,000 or even from 20,000 to 150,000.

Mixtures of polymers can also be used. This may in particular be beneficial to control the mechanical and/or dissolution properties of the compartment or pouch, depending on the application thereof and the required needs. For example, it may be preferred that a mixture of polymers is present in the material of the compartment, whereby one polymer material has a higher water-solubility than another polymer material, and/or one polymer material has a higher mechanical strength than another polymer material. It may be preferred that a mixture of polymers is used, having different weight average molecular weights, for example a mixture of PVA or a copolymer thereof of a weight average molecular weight of 10,000- 40,000, preferably around 20,000, and of PVA or copolymer thereof, with a weight average molecular weight of about 100,000 to 300,000, preferably around 150,000.

Also useful are polymer blend compositions, for example comprising hydrolytically degradable and water-soluble polymer blend such as polylactide and polyvinyl alcohol, achieved by the mixing of polylactide and polyvinyl alcohol,

typically comprising 1-35% by weight polylactide and approximately from 65% to 99% by weight polyvinyl alcohol, if the material is to be water-dispersible, or water-soluble.

It may be preferred that the polymer present in the film is from 60 to 98% hydrolysed, preferably 80% to 90% hydrolysed, to improve the dissolution of the material.

Most preferred are films which are water-soluble and stretchable films, as described above. Highly preferred water-soluble films are films which comprise PVA polymers and that have similar properties to the film known under the trade reference Monosol®8630, as sold by Chris-Craft Industrial Products of Gary, Indiana, US and also PT-75, as sold by Aicello of Japan.

The water-soluble film herein may comprise other additive ingredients than the polymer or polymer material. For example, it may be beneficial to add plasticisers, for example glycerol, ethylene glycol, diethylene glycol, propylene glycol, sorbitol and mixtures thereof, additional water, disintegrating aids. It may be useful that the pouch or water-soluble film itself comprises a detergent additive to be delivered to the wash water, for example organic polymeric soil release agents, dispersants, dye transfer inhibitors.

The pouch is typically made by a process comprising the steps of contacting a composition herein to a water-soluble film in such a way as to partially enclose the composition to obtain a partially formed pouch. The composition may already contain at least one solid particle per pouch, or, alternatively, one or more of the solid particles may be added at this stage of the process. Optionally the first water-soluble film of the partially formed pouch is then contacted with a second water-soluble film, and the films are sealed together to provide the fully formed pouch. The first and second water-soluble films may be identical in terms of material specifications and physical properties (e.g. thickness), but this need not necessarily be the case.

In a preferred process, the pouch is made using a mold, preferably the mold has round inner side walls and a round inner bottom wall. A liquid medium and at least one solid particle may then be transferred into the mould, a second water-

soluble film may be placed over the mould with the composition and the pouch may then be sealed. Preferably the first and second films are sealed by heat sealing or by solvent sealing. The film is preferably stretched during the formation of the pouch. Suitable pouch-forming processes are disclosed in US-A-3 218 776, issued on 23rd November 1965, and assigned to Cloud Machine Corp.

Liquid Composition

Typically the liquid composition is contained in the inner volume space of the pouch, and it may be divided over one or more compartments of the pouch.

The liquid composition preferably has a density of 0.8kg/l to 1.3kg/l, preferably about 1.0 to 1.1 kg/l. The liquid composition can be made by any method and can have any viscosity, typically depending on its ingredients. The viscosity may be controlled, if desired, by using various viscosity modifiers such as hydrogenated castor oil and/or solvents. Hydrogenated castor oil is commercially available as Thixcin®. Suitable solvents are described in more detail below.

The liquid compositions of the present invention are concentrated and contain low levels of water. The liquid compositions comprise less than 10% by weight water, and preferably less than 6% by weight water. Suitable compositions may even comprise less than 4% by weight water.

The compositions herein are typically cleaning compositions or fabric care compositions, preferably hard surface cleaners, more preferably laundry or dish washing compositions, including pre-treatment or soaking compositions and rinse additive compositions, including fabric enhancers such as softeners, anti-wrinkling agents, perfume compositions. Particularly preferred are fabric cleaning compositions (laundry detergents).

Preferred ingredients of the liquid composition

The preferred amounts of ingredients described herein are % by weight of the composition herein as a whole.

If the liquid composition is a detergent composition, it is preferred that at least a surfactant and builder are present, preferably at least anionic surfactant and preferably also nonionic surfactant, and preferably at least a builder, more

preferably at least a water-soluble builder such as phosphate builder and/or fatty acid builder.

Other preferred components are enzymes and/or bleaching agents, such as a preformed peroxyacid.

Highly preferred are also perfume, brightener, buffering agents (to maintain the pH preferably from 5.5 to 9, more preferably 6 to 8), fabric softening agents, including clays and silicones benefit agents, suds suppressors.

In hard-surface cleaning compositions and dish wash compositions, it is preferred that at least a water-soluble builder is present, such as a phosphate, and preferably also surfactant, perfume, enzymes, bleach.

In fabric enhancing compositions, preferably at least a perfume and a fabric benefit agent are present for example a cationic softening agent, or clay softening agent, anti-wrinkling agent, fabric substantive dye.

Highly preferred in all above compositions are also additional solvents, such as alcohols, diols, monoamine derivatives, glycerol, glycols, polyalkylene glycols, such as polyethylene glycol. Highly preferred are mixtures of solvents, such as mixtures of alcohols, mixtures of diols and alcohols, mixtures. Highly preferred may be that (at least) an alcohol, diol, monoamine derivative and preferably even glycerol are present. The compositions of the invention are preferably concentrated liquids having preferably less than 50% or even less than 40% by weight of solvent (other than water), preferably less than 30% or even less than 20% or even less than 35% by weight. Preferably the solvent is present at a level of at least 5% or even at least 10% or even at least 15% by weight of the composition.

Highly preferred is that the composition comprises a plasticiser for the water-soluble pouch material, for example one of the plasticisers described above, for example glycerol. Such plasticisers can have the dual purpose of being a solvent for the other ingredients of the composition and a plasticiser for the pouch material.

Anionic Surfactant

The detergent compositions of the invention comprise preferably a surfactant system. Preferably, at least an anionic surfactant is present, preferably at least an sulphonic acid surfactant, such as a linear alkyl benzene sulphonic acid, but salt forms may also be used. Preferably, at least 15% or even at least 20% or even at least 30% by weight of the composition is a surfactant, up to 70% or even 60% or even 50% by weight. Preferably, at least an anionic surfactant and an nonionic surfactant are present in the surfactant system of the composition, preferably in a ratio of 1:2 to 2:1, and more preferably 1.5:1 to 1:1.5.

The anionic surfactant(s), are preferably present at a level of at least 7.5% by weight of the composition. More preferably anionic surfactant is present at a level of from 10% or even at least 15%, or even from 22.5% by weight of the composition.

Anionic sulfonate or sulfonic acid surfactants suitable for use herein include the acid and salt forms of a C₅-C₂₀, more preferably a C₁₀-C₁₆, more preferably a C₁₁-C₁₃ alkylbenzene sulfonates, alkyl ester sulfonates, C₆-C₂₂ primary or secondary alkane sulfonates, sulfonated polycarboxylic acids, and any mixtures thereof, but preferably C₁₁-C₁₃ alkylbenzene sulfonates.

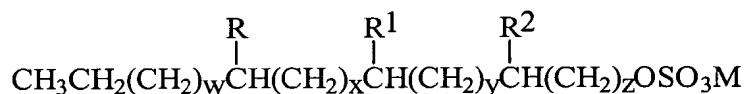
Anionic sulphate salts or acids surfactants suitable for use in the compositions of the invention include the primary and secondary alkyl sulphates, having a linear or branched alkyl or alkenyl moiety having from 9 to 22 carbon atoms or more preferably C₁₂ to C₁₈ alkyl.

Highly preferred are beta-branched alkyl sulphate surfactants or mixtures of commercial available materials, having a weight average (of the surfactant or the mixture) branching degree of at least 50% or even at least 60% or even at least 80% or even at least 95%. It has been found that these branched sulphate surfactants provide a much better viscosity profile, when clays are present, particular when 5% or more clay is present.

It may be preferred that the only sulphate surfactant is such a highly branched alkyl sulphate surfactant. Accordingly only one type of commercially available branched alkyl sulphate surfactant is present, whereby the weight average

branching degree is at least 50%, preferably at least 60% or even at least 80%, or even at least 90%. Preferred is for example Isalchem®, as available from Condea.

Mid-chain branched alkyl sulphates or sulfonates are also suitable anionic surfactants for use in the compositions of the invention. Preferred are the mid-chain branched alkyl sulphates. Preferred mid-chain branched primary alkyl sulphate surfactants are of the formula



These surfactants have a linear primary alkyl sulphate chain backbone (i.e., the longest linear carbon chain which includes the sulphated carbon atom), which preferably comprises from 12 to 19 carbon atoms and their branched primary alkyl moieties comprise preferably a total of at least 14 and preferably no more than 20, carbon atoms. In compositions or components thereof of the invention comprising more than one of these sulphate surfactants, the average total number of carbon atoms for the branched primary alkyl moieties is preferably within the range of from greater than 14.5 to about 17.5. Thus, the surfactant system preferably comprises at least one branched primary alkyl sulphate surfactant compound having a longest linear carbon chain of not less than 12 carbon atoms or not more than 19 carbon atoms, and the total number of carbon atoms including branching must be at least 14, and further the average total number of carbon atoms for the branched primary alkyl moiety is within the range of greater than 14.5 to about 17.5.

Preferred mono-methyl branched primary alkyl sulphates are selected from the group consisting of: 3-methyl pentadecanol sulphate, 4-methyl pentadecanol sulphate, 5-methyl pentadecanol sulphate, 6-methyl pentadecanol sulphate, 7-methyl pentadecanol sulphate, 8-methyl pentadecanol sulphate, 9-methyl pentadecanol sulphate, 10-methyl pentadecanol sulphate, 11-methyl pentadecanol sulphate, 12-methyl pentadecanol sulphate, 13-methyl pentadecanol sulphate, 3-methyl hexadecanol sulphate, 4-methyl hexadecanol sulphate, 5-methyl hexadecanol sulphate, 6-methyl hexadecanol sulphate, 7-methyl hexadecanol sulphate, 8-methyl hexadecanol sulphate, 9-methyl

hexadecanol sulphate, 10-methyl hexadecanol sulphate, 11-methyl hexadecanol sulphate, 12-methyl hexadecanol sulphate, 13-methyl hexadecanol sulphate, 14-methyl hexadecanol sulphate, and mixtures thereof.

Preferred di-methyl branched primary alkyl sulphates are selected from the group consisting of: 2,3-methyl tetradecanol sulphate, 2,4-methyl tetradecanol sulphate, 2,5-methyl tetradecanol sulphate, 2,6-methyl tetradecanol sulphate, 2,7-methyl tetradecanol sulphate, 2,8-methyl tetradecanol sulphate, 2,9-methyl tetradecanol sulphate, 2,10-methyl tetradecanol sulphate, 2,11-methyl tetradecanol sulphate, 2,12-methyl tetradecanol sulphate, 2,3-methyl pentadecanol sulphate, 2,4-methyl pentadecanol sulphate, 2,5-methyl pentadecanol sulphate, 2,6-methyl pentadecanol sulphate, 2,7-methyl pentadecanol sulphate, 2,8-methyl pentadecanol sulphate, 2,9-methyl pentadecanol sulphate, 2,10-methyl pentadecanol sulphate, 2,11-methyl pentadecanol sulphate, 2,12-methyl pentadecanol sulphate, 2,13-methyl pentadecanol sulphate, and mixtures thereof.

It is preferred that the anionic surfactants herein are present in the form of sodium salts.

Nonionic alkoxyated surfactant

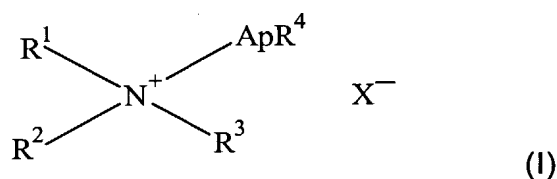
Ethoxylated and propoxylated nonionic surfactants are preferred. Preferred alkoxyated surfactants can be selected from the classes of the nonionic condensates of alkyl phenols, nonionic ethoxylated alcohols, nonionic ethoxylated/propoxylated fatty alcohols.

Highly preferred are nonionic alkoxyated alcohol surfactants, being the condensation products of aliphatic alcohols with from 1 to 75 moles of alkylene oxide, in particular about 50 or from 1 to 15 moles, preferably to 11 moles, particularly ethylene oxide and/or propylene oxide, are highly preferred nonionic surfactants. The alkyl chain of the aliphatic alcohol can either be straight or branched, primary or secondary, and generally contains from 6 to 22 carbon atoms. Particularly preferred are the condensation products of alcohols having an alkyl group containing from 8 to 20 carbon atoms with from 2 to 9 moles and in particular 3 or 5 moles, of ethylene oxide per mole of alcohol.

Polyhydroxy fatty acid amides are highly preferred nonionic surfactant comprised by the composition, in particular those having the structural formula R^2CONR^1Z wherein : R^1 is H, C_1 -18, preferably C_1 - C_4 hydrocarbyl, 2-hydroxy ethyl, 2-hydroxy propyl, ethoxy, propoxy, or a mixture thereof, preferable C_1 - C_4 alkyl, more preferably C_1 or C_2 alkyl, most preferably C_1 alkyl (i.e., methyl); and R^2 is a C_5 - C_{31} hydrocarbyl, preferably straight-chain C_5 - C_{19} or C_7 - C_{19} alkyl or alkenyl, more preferably straight-chain C_9 - C_{17} alkyl or alkenyl, most preferably straight-chain C_{11} - C_{17} alkyl or alkenyl, or mixture thereof; and Z is a polyhydroxyhydrocarbyl having a linear hydrocarbyl chain with at least 3 hydroxyls directly connected to the chain, or an alkoxyated derivative (preferably ethoxylated or propoxylated) thereof. Z preferably will be derived from a reducing sugar in a reductive amination reaction; more preferably Z is a glycityl.

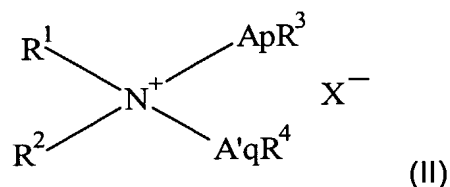
Cationic surfactant

Suitable cationic mono-alkoxylated and bis-alkoxylated quaternary amine surfactants with a C_6 - C_{18} N-alkyl chain, such as of the general formula I:



wherein R^1 is an alkyl or alkenyl moiety containing from about 6 to about 18 carbon atoms, preferably 6 to about 16 carbon atoms, most preferably from about 6 to about 14 carbon atoms; R^2 and R^3 are each independently alkyl groups containing from one to about three carbon atoms, preferably methyl, most preferably both R^2 and R^3 are methyl groups; R^4 is selected from hydrogen (preferred), methyl and ethyl; X^- is an anion such as chloride, bromide, methylsulphate, sulphate, or the like, to provide electrical neutrality; A is a alkoxy group, especially a ethoxy, propoxy or butoxy group; and p is from 0 to about 30, preferably 2 to about 15, most preferably 2 to about 8.

The cationic bis-alkoxylated amine surfactant preferably has the general formula II:



wherein R^1 is an alkyl or alkenyl moiety containing from about 8 to about 18 carbon atoms, preferably 10 to about 16 carbon atoms, most preferably from about 10 to about 14 carbon atoms; R^2 is an alkyl group containing from one to three carbon atoms, preferably methyl; R^3 and R^4 can vary independently and are selected from hydrogen (preferred), methyl and ethyl, X^- is an anion such as chloride, bromide, methylsulphate, sulphate, or the like, sufficient to provide electrical neutrality. A and A' can vary independently and are each selected from C_1 - C_4 alkoxy, especially ethoxy, (i.e., $-\text{CH}_2\text{CH}_2\text{O}-$), propoxy, butoxy and mixtures thereof; p is from 1 to about 30, preferably 1 to about 4 and q is from 1 to about 30, preferably 1 to about 4, and most preferably both p and q are 1.

Another suitable group of cationic surfactants which can be used in the detergent compositions are cationic ester surfactants. Suitable cationic ester surfactants, including choline ester surfactants, have for example been disclosed in US Patents No.s 4228042, 4239660 and 4260529.

Builder compounds

The compositions in accord with the present invention preferably contain a water-soluble builder compound, typically present in detergent compositions at a level of from 1% to 60% by weight, preferably from 3% to 40% by weight, most preferably from 5% to 25% by weight of the composition.

Suitable water-soluble builder compounds include the water soluble monomeric carboxylates, or their acid forms, or homo or copolymeric polycarboxylic acids or their salts in which the polycarboxylic acid comprises at least two carboxylic radicals separated from each other by not more than two carbon atoms, and mixtures of any of the foregoing.

Highly preferred maybe that one or more fatty acids and/ or optionally salts thereof (and then preferably sodium salts) are present in the detergent composition. It has been found that this can provide further improved softening and cleaning of the fabrics. Preferably, the compositions contain 1% to 25% by weight of a fatty acid or salt thereof, more preferably 6% to 18% or even 10% to 16% by weight. Preferred are in particular C₁₂-C₁₈ saturated and/ or unsaturated fatty acids, but preferably mixtures of such fatty acids. Highly preferred have been found mixtures of saturated and unsaturated fatty acids, for example preferred is a mixture of rape seed-derived fatty acid and C₁₆-C₁₈ topped whole cut fatty acids, or a mixture of rape seed-derived fatty acid and a tallow alcohol derived fatty acid.

The detergent compositions of the invention may comprise phosphate-containing builder material. Preferably present at a level of from 2% to 40%, more preferably from 3% to 30%, more preferably from 5% to 20%. Suitable examples of water-soluble phosphate builders are the alkali metal triphosphates, sodium, potassium and ammonium pyrophosphate, sodium and potassium and ammonium pyrophosphate, sodium and potassium orthophosphate, sodium polymeta/phosphate in which the degree of polymerization ranges from about 6 to 21, and salts of phytic acid.

The compositions in accord with the present invention may contain a partially soluble or insoluble builder compound, typically present in detergent compositions at a level of from 0.5% to 60% by weight, preferably from 5% to 50% by weight, most preferably from 8% to 40% weight of the composition. Preferred are aluminosilicates, such as Zeolite A or zeolite MAP and/or crystalline layered silicates such as SKS-6®, available from Clariant.

However, from a formulation point of view it may be preferred not to include such builders in the liquid composition, because it will lead to too much dispersed or precipitate material in the liquid, or it requires too much process or dispersion aids.

Chelating agents

The composition may comprise a chelating agent, typically a high ionic strength chelating agent, having two or more phosphonic acid or phosphonate groups, or

two or more carboxylic acid or carboxylate groups, or mixtures thereof. By chelating agent it is meant herein components which act to sequester (chelate) heavy metal ions, but these components may also have calcium and magnesium chelation capacity.

Chelating agents are generally present at a level of from 1%, preferably from 2.5% from 3.5% or even 5.0% or even 7% and preferably up to 20% or even 15% or even 10% by weight of the composition herein.

Highly suitable organic phosphonates herein are amino alkylene poly (alkylene phosphonates), alkali metal ethane 1-hydroxy bisphosphonates and nitrilo trimethylene phosphonates. Preferred among the above species are diethylene triamine penta (methylene phosphonate), ethylene diamine tri (methylene phosphonate) hexamethylene diamine tetra (methylene phosphonate) and hydroxy-ethylene 1,1 diphosphonate.

Other suitable chelating agents for use herein include nitrilotriacetic acid and polyaminocarboxylic acids such as ethylenediaminetetracetic acid, ethylenetriamine pentacetic acid, ethylenediamine disuccinic acid, ethylenediamine diglutamic acid, 2-hydroxypropylenediamine disuccinic acid or any salts thereof. Especially preferred is ethylenediamine-N,N'-disuccinic acid (EDDS) or the alkali metal, alkaline earth metal, ammonium, or substituted ammonium salts thereof, or mixtures thereof.

Glycinamide-N,N'-disuccinic acid (GADS), ethylenediamine-N,N'-diglutamic acid (EDDG) and 2-hydroxypropylenediamine-N,N'-disuccinic acid (HPDDS) are also suitable.

Suitable chelating agents with two or more carboxylates or carboxylic acid groups include the acid or salt forms of succinic acid, malonic acid, (ethylenedioxy) diacetic acid, maleic acid, diglycolic acid, tartaric acid, tartronic acid and fumaric acid, as well as the ether carboxylates and the sulfinyl carboxylates. Chelants containing three carboxy groups include, in particular, the acids or salt forms of citrates, aconitrates and citraconates as well as succinate derivatives. Preferred carboxylate chelants are hydroxycarboxylates containing up to three carboxy groups per molecule, more particularly citrates and citric acids.

Chelating agents containing four carboxy groups include the salts and acid forms of oxydisuccinates, 1,1,2,2-ethane tetracarboxylates, 1,1,3,3-propane tetracarboxylates and 1,1,2,3-propane tetracarboxylates, sulfosuccinate derivatives.

Highly preferred it that at least one organo phosphonate or phosphonic acid and also at least one di- or tri-carboxylate or carboxylic acid is present. Highly preferred is that at least fumaric acid (or salt) and citric acid (or salt) and one or more phosphonates are present. Preferred salts are sodium salts.

Perfume

Highly preferred are perfume components, preferably at least one component comprising a coating agent and/ or carrier material, preferably organic polymer carrying the perfume or aluminosilicate carrying the perfume, or an encapsulate enclosing the perfume, for example starch or other cellulosic material encapsulate. In a particularly preferred embodiment of the present invention the solid particle is a perfume encapsulate.

Fabric softening clays

Preferred fabric softening clays are smectite clays, which can also be used to prepare the organophilic clays described hereinafter, for example as disclosed in EP-A-299575 and EP-A-313146. Specific examples of suitable smectite clays are selected from the classes of the bentonites- also known as montmorillonites, hectorites, volchonskoites, nontronites, saponites and sauconites, particularly those having an alkali or alkaline earth metal ion within the crystal lattice structure.

Preferably, hectorites or montmorillonites or mixtures thereof. Hectorites are most preferred clays.

The softening clay if present, may be used at levels up to about 15%, more preferably from about 3% to about 10% by weight, when the formulation is to be a fabric softening formulation.

The hectorite clays suitable in the present composition should preferably be sodium clays, for better softening activity. Sodium clays are either naturally occurring, or are naturally-occurring calcium-clays which have been treated so as to convert them to sodium-clays. If calcium-clays are used in the present compositions, a salt of sodium can be added to the compositions in order to convert the calcium clay to a sodium clay. Preferably, such a salt is sodium carbonate, typically added at levels of up to 5% of the total amount of clay. Examples of hectorite clays suitable for the present compositions include Bentone EW® as sold by Elementis.

Another preferred clay is an organophilic clay, preferably a smectite clay, whereby at least 30% or even at least 40% or preferably at least 50% or even at least 60% of the exchangeable cations is replaced by a, preferably long-chain, organic cations. Such clays are also referred to as hydrophobic clays. The cation exchange capacity of clays and the percentage of exchange of the cations with the long-chain organic cations can be measured in several ways known in the art, as for example fully set out in Grimshaw, *The Chemistry and Physics of Clays*, Interscience Publishers, Inc., pp. 264-265 (1971).

Whilst the organophilic smectite clay provides excellent softening benefit, they can increase the viscosity of the liquid compositions. Therefore, it will depend on the viscosity requirements of the composition, how much of these organophilic clays can be used. Typically, they are used in the liquid detergent compositions of the invention at a level of from 0.1% to 10%, more preferably from 0.3% to 7%, most preferably from 0.4% to 5% or even 0.5% to 4% by weight of the composition.

These organophilic clays are formed prior to incorporation into the detergent composition. Thus for example, the cations, or part thereof, of the normal smectite clays are replaced by the long-chain organic cations to form the organophilic smectite clays herein, prior to further processing of the material to form the detergents of the invention.

The organophilic clay is preferably in the form of a platelet or lath-shaped particle. Preferably the ratio of the width to the length of such a platelet is at least 1:2, preferably at least 1:4 or even at least 1:6 or even at least 1:8.

When used herein, a long-chain organic cation can be any compound which comprises at least one chain having at least 6 carbon atoms, but typically at least 10 carbon atoms, preferably at least 12 carbon atoms, or in certain embodiments of the invention, at least 16 or even at least 18 carbon atoms. Preferred long-chain organic cations are described hereinafter.

Preferred organophilic clays herein are smectite clays, preferably hectorite clays and/ or montmorillonite clays containing one or more organic cations of formulae:



where R_1 represents an organic radical selected from R_7 , $R_7\text{-CO-O-(CH}_2)_n$, or

$R_7\text{-CO-NR}_8$ in which R_7 is an alkyl, alkenyl or alkylaryl group with 12-22 carbon atoms, whereby R_8 is hydrogen, C_1 - C_4 alkyl, alkenyl or hydroxyalkyl, preferably - CH_3 or - C_2H_5 or -H; n is an integer, preferably equal to 2 or 3; R_2 represents an organic radical selected from R_1 or C_1 - C_4 alkyl, alkenyl or hydroxyalkyl, preferably - CH_3 or - CH_2CH_2OH ; R_3 and R_4 are organic radicals selected from C_1 - C_4 alkyl-aryl, C_1 - C_4 alkyl, alkenyl or hydroxyalkyl, preferably - CH_3 , - CH_2CH_2OH , or benzyl group; R_5 is an alkyl or alkenyl group with 12-22 carbon atoms; R_6 is preferably -OH, - $NHCO\text{-}R_7$, or - $OCO\text{-}R_7$.

Highly preferred cations are quaternary ammonium cations having two C_{16} - C_{28} or even C_{16} - C_{24} alkyl chains. Highly preferred are one or more organic cations which have one or preferably two alkyl groups derived from natural fatty alcohols, the cations preferably being selected from dicocoyl methyl benzyl ammonium, dicocoyl ethyl benzyl ammonium, dicocoyl dimethyl ammonium, dicocoyl diethyl ammonium; more preferably ditallow diethyl ammonium, ditallow ethyl benzyl ammonium; more preferably ditallow dimethyl ammonium and/ or ditallow methyl benzyl ammonium.

It may be highly preferred that mixtures of organic cations are present.

Highly preferred are organophilic clays as available from Rheox/Elementis, such as Bentone SD-1 and Bentone SD-3, which are registered trademarks of Rheox/Elementis.

In a particularly preferred embodiment of the present invention the solid particle is a clay particle, such as a clay agglomerate or extrudate.

Cationic fabric softening agents

Cationic fabric softening agents are preferably present in the composition herein. Suitable cationic fabric softening agents include the water insoluble tertiary amines or dilong chain amide materials as disclosed in GB-A-1 514 276 and EP-B-0 011 340. Preferably, these water-insoluble tertiary amines or dilong chain amide materials are comprised by the solid component of the composition herein.

Cationic fabric softening agents are typically incorporated at total levels of from 0.5% to 15% by weight, normally from 1% to 5% by weight.

Bleaching agent

Another ingredient which may be present is a perhydrate bleach, such as salts of percarbonates, particularly the sodium salts, and/ or organic peroxyacid bleach precursor. It has been found that when the pouch or compartment is formed from a material with free hydroxy groups, such as PVA, the preferred bleaching agent comprises a percarbonate salt and is preferably free from any perborate salts or borate salts. It has been found that borates and perborates interact with these hydroxy-containing materials and reduce the dissolution of the materials and also result in reduced performance.

Inorganic perhydrate salts are a preferred source of peroxide. Preferably these salts are present at a level of from 0.01% to 50% by weight, more preferably of from 0.5% to 30% by weight of the composition or component.

Examples of inorganic perhydrate salts include percarbonate, perphosphate, persulfate and persilicate salts. The inorganic perhydrate salts are normally the alkali metal salts. The inorganic perhydrate salt may be included as the crystalline solid without additional protection. For certain perhydrate salts

however, the preferred executions of such granular compositions utilise a coated form of the material which provides better storage stability for the perhydrate salt in the granular product. Suitable coatings comprise inorganic salts such as alkali metal silicate, carbonate or borate salts or mixtures thereof, or organic materials such as waxes, oils, or fatty soaps.

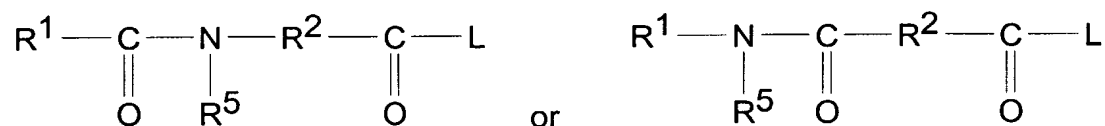
Alkali metal percarbonates, particularly sodium percarbonate are preferred perhydrates herein. Sodium percarbonate is an addition compound having a formula corresponding to $2\text{Na}_2\text{CO}_3 \cdot 3\text{H}_2\text{O}_2$, and is available commercially as a crystalline solid.

The composition herein preferably comprises a peroxy acid or a precursor therefor (bleach activator), preferably comprising an organic peroxyacid bleach precursor. It may be preferred that the composition comprises at least two peroxy acid bleach precursors, preferably at least one hydrophobic peroxyacid bleach precursor and at least one hydrophilic peroxy acid bleach precursor, as defined herein. The production of the organic peroxyacid occurs then by an in situ reaction of the precursor with a source of hydrogen peroxide.

The hydrophobic peroxy acid bleach precursor preferably comprises a compound having a oxy-benzene sulphonate group, preferably NOBS, DOBS, LOBS and/or NACA-OBS, as described herein.

The hydrophilic peroxy acid bleach precursor preferably comprises TAED, as described herein.

Amide substituted alkyl peroxyacid precursor compounds are suitable herein, including those of the following general formulae:

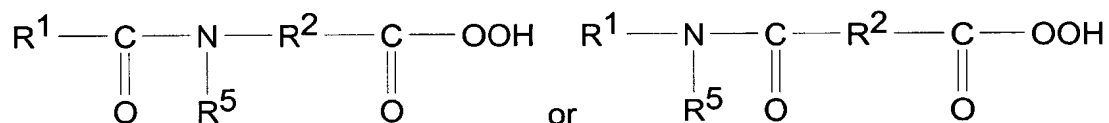


wherein R^1 is an alkyl group with from 1 to 14 carbon atoms, R^2 is an alkylene group containing from 1 to 14 carbon atoms, and R^5 is H or an alkyl group containing 1 to 10 carbon atoms and L can be essentially any leaving group.

Amide substituted bleach activator compounds of this type are described in EP-A-0 170 386.

The composition may contain a pre-formed organic peroxyacid.

A preferred class of organic peroxyacid compounds are the amide substituted compounds of the following general formulae:



wherein R¹ is an alkyl, aryl or alkaryl group with from 1 to 14 carbon atoms, R² is an alkylene, arylene, and alkarylene group containing from 1 to 14 carbon atoms, and R⁵ is H or an alkyl, aryl, or alkaryl group containing 1 to 10 carbon atoms. Amide substituted organic peroxyacid compounds of this type are described in EP-A-0 170 386.

Other organic peroxyacids include diacyl and tetraacylperoxides, especially diperoxydodecanedioic acid, diperoxytetradecanedioic acid and diperoxyhexadecanedioic acid. Mono- and diperazelaic acid, mono- and diperbrassylic acid and N-phthaloylaminoperoxicaproic acid are also suitable herein.

In a particularly preferred embodiment of the present invention the solid particle is a particulate bleach or bleach activator.

Suds suppressing system

The composition may comprise a suds suppresser at a level less than 10%, preferably 0.001% to 10%, preferably from 0.01% to 8%, most preferably from 0.05% to 5%, by weight of the composition. Preferably the suds suppresser is either a soap, paraffin, wax, or any combination thereof. If the suds suppresser is a suds suppressing silicone, then the detergent composition preferably comprises from 0.005% to 0.5% by weight a suds suppressing silicone.

Enzymes

Another preferred ingredient useful in the compositions herein is one or more enzymes.

Preferred enzymatic materials include the commercially available lipases, cutinases, amylases, neutral and alkaline proteases, cellulases, endolases, esterases, pectinases, lactases and peroxidases conventionally incorporated into detergent compositions. Suitable enzymes are discussed in US Patents 3,519,570 and 3,533,139.

Preferred commercially available protease enzymes include those sold under the tradenames Alcalase, Savinase, Primase, Durazym, and Esperase by Novo Industries A/S (Denmark), those sold under the tradename Maxatase, Maxacal and Maxapem by Gist-Brocades, those sold by Genencor International, and those sold under the tradename Opticlean and Optimase by Solvay Enzymes. Protease enzyme may be incorporated into the compositions in accordance with the invention at a level of from 0.0001% to 4% active enzyme by weight of the composition.

Preferred amylases include, for example, α -amylases obtained from a special strain of *B licheniformis*, described in more detail in GB-1,269,839 (Novo). Preferred commercially available amylases include for example, those sold under the tradename Rapidase by Gist-Brocades, and those sold under the tradename Termamyl, Duramyl and BAN by Novo Industries A/S. Highly preferred amylase enzymes maybe those described in PCT/ US 9703635, and in WO95/26397 and WO96/23873.

Amylase enzyme may be incorporated into the composition in accordance with the invention at a level of from 0.0001% to 2% active enzyme by weight of the composition.

In a particularly preferred embodiment of the present invention the solid particle is an enzyme encapsulate.

Useful additional non-alkoxylated organic polymeric compounds for inclusion in the compositions herein include the water soluble organic homo- or co-polymeric

polycarboxylic acids or their salts in which the polycarboxylic acid comprises at least two carboxyl radicals separated from each other by not more than two carbon atoms. Polymers of the latter type are disclosed in GB-A-1,596,756. Examples of such salts are polyacrylates of MWt 1000-5000 and their copolymers with maleic anhydride, such copolymers having a molecular weight of from 2000 to 100,000, especially 40,000 to 80,000.

Other organic polymeric compounds suitable for incorporation in the detergent compositions herein include cellulose derivatives.

Suitable suds suppressing systems for use herein may comprise essentially any known antifoam compound, including for example silicone antifoam compounds and 2-alkyl alanol antifoam compounds.

Other suitable antifoam compounds include the monocarboxylic fatty acids and soluble salts thereof, as also described as builders above. These materials are described in US Patent 2,954,347, issued September 27, 1960 to Wayne St. John. The monocarboxylic fatty acids, and salts thereof, for use as suds suppressor typically have hydrocarbyl chains of 10 to 24 carbon atoms, preferably 12 to 18 carbon atoms. Suitable salts include the alkali metal salts such as in particular sodium but also potassium salts.

The compositions herein may also comprise from 0.01% to 10 %, preferably from 0.05% to 0.5% by weight of polymeric dye transfer inhibiting agents. The polymeric dye transfer inhibiting agents are preferably selected from polyamine N-oxide polymers, copolymers of N-vinylpyrrolidone and N-vinylimidazole, polyvinylpyrrolidone polymers or combinations thereof, whereby these polymers can be cross-linked polymers.

The compositions herein also optionally contain from about 0.005% to 5% by weight of certain types of hydrophilic optical brighteners. Preferred brighteners include 4,4',-bis[(4-anilino-6-(N-2-bis-hydroxyethyl)-s-triazine-2-yl)amino]-2,2'-stilbenedisulfonic acid and disodium salt, commercially marketed under the tradename Tinopal-UNPA-GX by Ciba-Geigy Corporation; 4,4'-bis[(4-anilino-6-(N-2-hydroxyethyl-N-methylamino)-s-triazine-2-yl)amino]2,2'-stilbenedisulfonic acid disodium salt, commercially marketed under the tradename Tinopal 5BM-GX by

Ciba-Geigy Corporation; 4,4'-bis[(4-anilino-6-morpholino-s-triazine-2-yl)amino]2,2'-stilbenedisulfonic acid, sodium salt, commercially marketed under the tradename Tinopal-DMS-X and Tinopal AMS-GX by Ciba Geigy Corporation.

Also preferred may be bleaches, neutralizing agents, buffering agents, phase regulants, hydrotropes, enzyme stabilizing agents, opacifiers, anti-oxidants, bactericides, photo-bleaches.

Examples

Example 1

The following low-moisture liquid detergent media were prepared. Moisture level as made was about 3.8% (Formula 1A) and about 5.5% (Formula 1B).

ingredient	1A. weight percent	1B. weight percent
1,2-propanediol	15.7%	16.6%
Neodol® 23-9	18.7%	20.9%
C11.8 linear alkylbenzene sulfonic acid	22.0%	29.1%
formic acid	1.1%	1.0%
citric acid (50% soln.)	1.6%	2.3%
monoethanolamine	11.0%	10.4%
palm kernal fatty acid	16.6%	4.9%
sodium diethylenetriamine pentaacetate (40% soln.)	0.9%	0.3%
decyl amidopropylamine	1.8%	1.8%
polymer 1 (80%) ¹	1.6%	1.1%
polymer 2 (80%) ²	1.6%	2.3%
calcium formate (10% soln.)	-	0.06%
protease enzyme	2.5%	2.5%
amylase enzyme	0.3%	0.3%
brightener 15	0.2%	0.2%
dye	0.003%	0.003%
perfume	0.6%	0.8%
water	to balance	to balance

¹Polymer 1 is a polyethyleneimine (MW = 182) with av. degree of ethoxylation = 15.

²Polymer 2 is a polyethyleneimine (MW = 600) with av. degree of ethoxylation = 20.

Example 2

Uniform spherical particles containing a polymeric profragrance were prepared by adding a reaction product of δ -damascone and Lupasol® WF into molten Pluracol® E 4000 at 60°C. The melt was then cast into 10 mm spheres in a mold.

Ingredient	a	b
profragrance	6.0 %	6.0 %
Pluracol® E 4000	94.0 %	93.4 %
Expancel® 091DE50		0.6 %
weight of 10 mm diameter capsule	0.57 g	0.46 g

Samples a and b were placed in the low moisture liquid detergent media of Examples 1A and 1B and sealed in pouches of soluble polyvinyl alcohol film, Mono-Sol® 8630, (50 ml of detergent and one 10 mm capsule per pouch) to provide unitized dose liquid detergent compositions with visible fragrance capsules. The particles were stable in the low moisture detergent of Examples 1A and 1B and dissolved in the wash to impart a fresh scent to laundry after drying. The spherical particles of sample b are less dense than the detergent and float in the detergent in the pouch and rapidly dissolve when the pouch is added to the wash.

Example 3

Extruded particles containing sodium citrate were prepared by combining 54.4 g of Pluracol® E 4000 solution (48.7% in water), 204.1 g of sodium citrate dihydrate, and 41.2 g of water. The mixture was extruded through an 8 mm axial dye and cut into 8 mm lengths. After drying, the composition was:

ingredient	weight percent
Pluracol® E 4000	11.1 %
sodium citrate (as anhydrous)	74.8 %
water	14.1 %

Samples of the solid citrate particles were placed in the low moisture liquid detergent media of Examples 1A and 1B and sealed in pouches of soluble polyvinyl alcohol film, Mono-Sol® 8630, (50 ml of detergent and two solid particles per pouch) to provide unitized dose liquid detergent compositions with visible solid particles. The particles were visible and stable in the low moisture liquid detergent and rapidly dissolve when added to the wash.

Example 4

Uniform spherical particles containing an oxidation catalyst were prepared by combining dichloro(4,11-diethyl-1,4,8,11-tetraazabicyclo[6.6.2]hexadecane)manganese with molten Pluracol® E 4000 at 60°C followed by casting in a mold to give the following composition.

Ingredient	weight percent
bleach catalyst	6.0 %
Pluracol® E 4000	94.0 %
weight of 10 mm diameter capsule	0.59 g

Samples of the solid bleach catalyst particle were placed in the low moisture liquid detergent media of Examples 1A and 1B and sealed in pouches of soluble polyvinyl alcohol film, Mono-Sol® 8630, (50 ml of detergent and one solid particle per pouch) to provide unitized dose liquid detergent compositions with visible bleach catalyst particles and rapidly dissolve when added to the wash.

Example 5

Solid particles containing a fabric anti-abrasion agent and dye anti-fading agent were prepared by combining 25.0 g of modified cellulose, 5.60 g of solution of an imidazole-epichlorohydrin condensation oligomer (44.6% in H₂O), 20.8 g of sodium citrate dihydrate, 6.5 g of Acusol® 445N (45.0% in H₂O), and 38.5 g of H₂O, forming into particles approximately 10 mm in diameter, and drying. After drying the individual particles weighed approximately 0.6 g each and the composition was:

ingredient	weight percent
modified cellulose ³	44.5 %
imidazole-epichlorohydrin condensation oligomer ³	4.5 %
sodium citrate (as anhydrous)	32.4 %
sodium polyacrylate, Acusol® 445N	5.2 %
water	13.4 %

³ as described in WO 00/22078 A1

Samples of the solid particles were placed in the low moisture liquid detergent media of Examples 1A and 1B and sealed in pouches of soluble polyvinyl alcohol film, Mono-Sol® 8630, (50 ml of detergent) to provide unitized dose liquid detergent compositions with visible solid particles.

Example 6

In a further example coloured particles with a mean geometric diameter of a) 2mm and b) 5mm are made by adding dye onto starch base particles. These coloured particles or "speckles" are added to the liquid compositions of the previous examples for consumer desirable aesthetics. The speckles rapidly dissolve when added to the wash.

WHAT IS CLAIMED IS:

1. A liquid composition comprising a transparent or translucent liquid medium and solid particles contained within the liquid medium characterised in that the liquid medium comprises less than 10% by weight of water and the composition is contained within a pouch made from a transparent or translucent water-soluble material, so that the individual solid particles are visible from outside the pouch.
2. A liquid composition according to claim 1 wherein the water-soluble pouch material comprises water-soluble polymer selected from the group consisting of polyacrylates and water-soluble acrylate copolymers, methylcellulose, carboxymethylcellulose sodium, dextrin, ethylcellulose, hydroxyethyl cellulose, hydroxypropyl methylcellulose, maltodextrin, polymethacrylates and mixtures thereof.
3. A liquid composition according to claim 2 wherein the water-soluble pouch material comprises polyvinyl alcohol.
4. A liquid composition according to claim 1 wherein the solid particles have a mean geometric diameter of at least 0.5mm.
5. A liquid composition according to claim 4 wherein the solid particles have a mean geometric diameter of between 0.5mm and 12mm, preferably between 1mm and 5mm.
6. A liquid composition according to claim 1 wherein the solid particles comprise active components selected from the group consisting of bleach, bleach activator, enzymes, perfume and mixtures thereof.
7. A liquid composition according to any of the previous claims wherein at least some of the solid particles are coloured, non-white speckles.

8. A liquid composition according to claim 1 which is a unit dose of a laundry composition, the composition comprising at least i) anionic surfactant and ii) fatty acid.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 02/01500

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C11D17/04 C11D17/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 339 707 A (UNILEVER PLC ;UNILEVER NV (NL)) 2 November 1989 (1989-11-02) claims; examples ---	1-8
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A	GB 1 303 810 A (UNILEVER LIMITED) 24 January 1973 (1973-01-24) cited in the application claims; examples ---	1-8
A	GB 2 194 793 A (UNILEVER PLC) 16 March 1988 (1988-03-16) claims; examples ---	1-8
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☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

° Special categories of cited documents:

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- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *&* document member of the same patent family

Date of the actual completion of the international search

7 June 2002

Date of mailing of the international search report

17/06/2002

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INTERNATIONAL SEARCH REPORT

Ir..... Application No
PCT/US 02/01500

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 02/01500

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